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PATENT SPECIFICATION

NO DRAWINGS

Inventor: HAROLD APPELTON

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COMPLETE SPECIFICATION

Improvements in and relating to the Treatment of Phthalate Plasticiser Esters

- We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British Company, of Imperial Chemical House, Millbank, London, S.W.1., do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- This invention relates to the treatment of plasticiser esters.
- The esters of phthalic acid are widely used in plasticising polyvinyl chloride. When polyvinyl chloride compositions which include the esters are to be used as electrical insulation materials it is commonly desired that the plasticiser esters should have a high volume resistivity.
- According to this invention there is provided a process for increasing the electrical resistivity of a dialkyl ester of orthophthalic acid, which comprises contacting it at a temperature of 70—130°C with an adsorbent magnesium silicate.
- A particularly suitable temperature is about 100°C.
- Dialkyl esters of phthalic acid having from 4 to 20 carbon atoms in each alkyl group, and particularly such esters having from 7 to 14 carbon atoms in each alkyl group may be very effectively treated.
- It is preferred to use a hydrated magnesium silicate having a surface area greater than 1 square metre per gram and a number of commercial preparations are available. Magnesium trisilicate, for example as the B.P. preparation supplied in pulverised form, is a useful form of the material though it may be preferred to utilise more easily filterable preparations. Suitably also the synthetic hydrous magnesium silicate of formula $(\text{MgO} \cdot 2.5\text{SiO}_2 \cdot \text{H}_2\text{O})$ made according to US Patent No. 2,393,625 may be used. It is preferred that the magnesium silicate should comprise at least 50% of amorphous material.
- It is preferred to use at least 0.1% by weight of magnesium silicate compared with the ester though advantageously less than 5% is employed, for example from 0.2 to 1%.
- The treatment may be continued for as little as one or two minutes with beneficial effect, but it is preferred to continue the treatment for up to 10 minutes, and in cases where the best effects are desired much longer periods for example at most 1 hour may be employed. In general the higher the temperature the shorter the period of treatment required.
- The ester may be treated with magnesium silicate by percolation of the ester through a bed of magnesium silicate or by any other convenient means.
- Separations of the magnesium silicate from the ester may be achieved for example, by filtration for example through a kieselguhr bed.
- The invention is particularly applicable to the treatment of esters which have been made from alcohols and phthalic acid or anhydride using acids, for example sulphuric acid, as a catalyst.
- EXAMPLES
- Di-iso octyl phthalate prepared in the normal way from iso-octanol and phthalic acid in the presence of sulphuric acid as a catalyst was neutralised with NaOH, washed, dried under vacuum and placed in a glass flask equipped with a reflux condenser which was provided with heating means. The material was stirred with magnesium trisilicate BP and finally filtered through a bed of kieselguhr

supported on sintered glass. The conditions of treatment and the results are indicated below.

EXAMPLE 1

5 Three identical experiments were carried

out using 1% by weight of magnesium trisilicate compared with the ester at a temperature of 100°C. Treatment was continued for one hour.

INITIAL ESTER

Volume Resistivity	= 2.01×10^{11} ohm - cm at 24-25°C.
Acid Value	= 0.179 mgs. KOH/gm.
Colour	= 30 - 40 Hazen Units.

TREATED ESTER

Sample	Volume Resistivity at 24-25°C ohm - cm	Acid Value mgs KOH/gm	Colour Hazen Units
1	2.103×10^{12}	0.051	20 - 30
2	3.14×10^{12}	0.037	20 - 30
3	2.54×10^{12}	0.037	20 - 30

EXAMPLE 11

The effect of varying the concentration of magnesium trisilicate was investigated by con-

tacting the ester for a period of one hour at a temperature of 100°C with the quantities 15 of magnesium trisilicate indicated below.

INITIAL ESTER

Volume Resistivity	= 1.98×10^{11} ohm - cm at 24-25°C.
Acid Value	= 0.105 mgs. KOH/gm.
Colour	= 30 - 40 Hazen Units

TREATED ESTER

% w/w Magnesium Trisilicate on Ester	Volume Resistivity at 24-25°C ohm - cm	Acid Value mgs KOH/gm	Colour Hazen Units
0.1	5.84×10^{11}	0.088	20 - 30
0.5	9.34×10^{11}	0.077	20 - 30
1.0	2.35×10^{12}	0.047	20 - 30
2.0	2.70×10^{12}	0.036	15 - 20
3.0	1.70×10^{12}	0.051	20
4.0	2.76×10^{12}	0.025	15 - 20
5.0	1.11×10^{12}	0.041	15 - 20

EXAMPLE III

20 The rate of change of the volume resistivity and acid value of samples of the di-iso octyl

phthalate was investigated by heating the sample at 100°C in the presence of 0.5% by

weight of magnesium trisilicate. Samples were withdrawn at intervals, filtered and examined.

INITIAL ESTER

Volume Resistivity = 1.66×10^{11} ohm - cm at 24-25°C.

Acid Value = 0.176 mgs KOH/gm.

Time (mins)	Volume Resistivity at 24-25°C ohm - cm	Acid Value mgs KOH/gm
2	4.11×10^{11}	0.150
10	1.18×10^{12}	0.142
30	1.06×10^{12}	0.127
45	1.06×10^{12}	0.112
92	1.01×10^{12}	0.114
121	8.69×10^{11}	0.110
151	8.91×10^{11}	—

EXAMPLE IV

- The effect of temperature was investigated by treating the ester with 1% by weight of magnesium trisilicate BP for a period of one hour at different temperature and comparing the products. The results are given below.

INITIAL ESTER

Volume Resistivity = 1.66×10^{11} ohm - cm at 24-25°C.

Acid Value = 0.176 mgs KOH/gm.

Colour = 30 - 40 Hazen Units

TREATED ESTER

Temp. °C.	Volume Resistivity at 24-25°C ohm - cm	Acid Value mgs KOH/gm	Colour Hazen Units
100	2.35×10^{12}	0.089	10 - 15
125	2.09×10^{12}	0.104	5 - 10
165	5.96×10^{11}	0.245	10
182	6.04×10^{11}	0.276	20 - 30

10 WHAT WE CLAIM IS:—

1. A process for increasing the electrical resistivity of a dialkyl ester of orthophthalic acid which comprises contacting it with an adsorbent magnesium silicate at a temperature in the range 70—130°C.

2. A process as claimed in Claim 1 in which

each alkyl group has from 7 to 14 carbon atoms.

3. A process as claimed in either preceding claims in which the magnesium silicate is a hydrated magnesium silicate having a surface area of at least 1 square metre per gram.

4. A process as claimed in any preceding

- claim in which the magnesium silicate comprises at least 50% of amorphous material.
5. A process as claimed in any preceding claim which is carried out at about 100°C.
- 5 6. A process as claimed in any preceding claim in which from 0.2 to 1% by weight of the magnesium silicate, based on the weight of the ester of phthalic acid, is present.
7. A process as claimed in any preceding
- 10 claim in which the ester is contacted with the magnesium silicate for from two to ten minutes.
8. A process as claimed in any preceding claim which is carried out by percolating the ester through a bed of magnesium silicate. 15
9. A process as claimed in any preceding claim in which after the ester has been contacted with magnesium silicate, it is filtered through a bed of kieselguhr.
10. A process as claimed in any preceding claim whenever carried out substantially as described herein in the Examples. 20
11. Phthalate esters whenever treated by a process according to any preceding claim.
- WALTER SCOTT,
Agent for the Applicants.

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